

**DEVELOPMENT AND TESTING OF AN ADVANCED ACID
FRACTURE CONDUCTIVITY APPARATUS**

A Thesis

by

CHUNLEI ZOU

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of
MASTER OF SCIENCE

May 2006

Major Subject: Petroleum Engineering

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Approved by:

Chair of Committee,
Committee Members,

Head of Department,

Ding Zhu
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Stephen A. Holditch

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ABSTRACT

Development and Testing of an Advanced Acid
Fracture Conductivity Apparatus. (May 2006)

ChunLei Zou, B.S., Tsinghua University

Chair of Advisory Committee: Dr. Ding Zhu

Since the oil price has been stable at a high level, operators are trying to maximize their production to get maximum return of investment. To achieve this objective, all kinds of well stimulation technologies are applied to the proper candidate wells. Acid fracturing is a standard practice to increase the production rate and to improve ultimate recovery in carbonate reservoirs. There have been successful cases in most carbonate reservoirs around the world. However acid fracture performance varied significantly with the acid fluid type, pumping schedule, formation composition, rock embedment strength, reservoir pressure, and other downhole conditions. Engineers have tried to understand the acid transportation and dissolution mechanism and, wanted to optimize each acid job design and to predict the acid treatment effect.

We made an acid fracture conductivity apparatus capable of conducting acid fracturing experiments at conditions as close to the field treatment conditions as possible. With reliable laboratory experimental results, engineers will understand the acid fracturing mechanism and build a realistic model to improve the treatment design.

Our lab facility is customized for its tasks. The setup and experimental procedures are optimized to make the operations feasible and the results accurate. The fracture conductivity cell is per API standard and is modified to accommodate thick rock samples. The thick rock will create a similar downhole leakoff condition when acid flows across the fracture surface. The Chem/Meter pump is able to provide a pump rate that matches field operational conditions. All necessary measurements are recorded. The experimental data are processed and interpreted with statistics methodology.

Some preliminary acid fracture conductivity experiments were carried out. A few different types of fluids are used to investigate the effects of acid concentration, fluid

viscosity, and emulsification. All acid fluids had 15, 30 or 60 minutes contact time with carbonate rocks. The acid leakoff velocity is controlled at velocity 0.003~0.01 ft/min to simulate the downhole condition. Most of the experiments are successful. They can be used to validate an acid fracture conductivity model.

To my wife and my parents

ACKNOWLEDGMENTS

My advisors Dr. Ding Zhu and Dr. A. Daniel Hill guided me throughout the course of this research. I would like to express my acknowledgements to them.

Thanks also to Dr. Zhengdong Cheng, Maysam Pournik, and Frank Platt for their help and support in my graduate study. I also want to extend my gratitude to Schlumberger, ConocoPhillips, Chevron, Saudi Aramco and Petrobras, who provided funding for the acid fracturing research project.

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CHAPTER I

INTRODUCTION

1.1 Acid fracturing application

Acid fracturing is a standard practice to increase production rate and improve ultimate recovery in carbonate reservoirs (mostly limestone or dolomite formations). This technique was initially applied in oilfield in 1960s. It has been proven to be effective by applications in carbonate reservoirs around the world.

In acid fracturing treatment, viscous pad fluid and acid are injected in sequence using high pressure pumps at high rate which raise the bottom hole pressure higher than the formation fracture breakdown pressure and thus create fractures in the target zone. Acid is then transported through these fractures and penetrates further into formation rocks. During the same time, acid reacts with carbonate rocks on fracture surfaces and also creates wormholes into the formation. Due to rock heterogeneity and the random characteristic of chemical reactions, the reaction rates are not uniform along fracture surfaces. Thus the acid etches the fracture surfaces unevenly. After the treatment is finished, the fractures close under formation stress. However, there is remaining conductivity in closed fractures due to the unevenness on fractures surfaces.

The success of an acid fracturing treatment depends on the heterogeneous acid etching on formation fracture surfaces, the acid penetration length and rock embedment strength after the acid treatment.

Generic acid fracturing job procedure is:

1. Pump a viscous fluid pad at rates that exceed the formation breakdown pressure and create fractures in the rock.
2. Inject acid, which reacts with the rock as it penetrates into the formation.

This thesis follows the style of *Journal of Petroleum Technology*.

3. Inject displacement fluid after desired amount of acid is pumped.

Successful acid fracturing treatment creates fracture paths with good conductivity. The higher the fracture conductivity is, the better the stimulation result is. The fracture conductivity depends on the heterogeneous dissolution of the rock surface, the formation closure pressure and the acidized formation rock hardness. The acid penetration distance depends on the acid injection rate, acid leakoff rate and acid-rock reaction kinetics. The objective of an acid fracturing job design is to optimize the operational parameter (acid type, injection rate, pumping technique, etc.) and to maximize the stimulation ratio, basing on the knowledge about the reservoir and formation properties.

1.2 Review of previously published work

Acid fracturing treatment performance varies significantly with different formation composition, rock embedment strength, reservoir pressure, and other downhole conditions. Engineers have tried to understand acid transportation and dissolution mechanism, therefore to optimize acid fracturing job design, and to predict acid treatment results.

Researchers have conducted experimental and theoretical studies to reduce the uncertainty of acid fracture performance. Broaddus and Knox¹ did dynamic acid etching tests with different formation rocks and determined fracture flow capacity after acid reactions. They concluded that acid fracture flow capacity is a function of formation type, acid type and concentration, acid-rock contact time, and reaction temperature. They used the laboratory test results to assist computerized acid job design. The lab tests were helpful when optimizing treatment design for a specific formation. However, they were not able to develop a correlation between acid fracture flow capacity and acid treatment parameters, which might be applicable universally in acid fracturing design.

Barron et al.² measured acid reaction rates in a horizontal fracture model with smooth limestone walls. They conducted experiments over a wide range of fracture widths and acid flow rates, and then scaled up to predict the acid penetration distance.

Nierode and Kruk³ did experiments to study acid reaction with carbonate rocks on fracture surface and derived a kinetic model for the reaction. It was then used to design acid fracturing treatments, in conjunction with a fracture geometry model.

Gong⁴ studied acid fracturing with lab experiments and found a relationship of fracture conductivity with surface roughness and rock embedment strength. He developed a fracture deformation model to predict fracture conductivity with proper surface roughness, rock embedment strength and fracture closure pressure, which matched his experimental results.

Dong⁵ studied further with acid etching lab experiments and numerical modeling. He developed new acid etching correlation and validated it with his lab results. His work was focused on fractures network (naturally fractured reservoir). The same as all the previous work, his lab experiments was conducted at an injection rate less than or equal to 10 ml/min across the artificial fracture in his acid cell, which made the fluid flow Reynolds number is much smaller than field injection conditions.

1.3 Research objective

The objective of this research project is to design and build a lab facility for an advanced experimental study on acid fracturing conductivity. The apparatus will be used to carry out extensive experiments to unveil relationships between acid fracture conductivity and various parameters in acid fracturing treatments.

There are three stages for each set of acid fracture conductivity experiment: acid flow through a fracture between carbonate rocks; rock surface profile characterization after acid etching; acid fracture conductivity measurement under different closure pressure. The acid flow conditions are expected to mimic field acid fracturing job conditions, which means that acid flowrate is high enough to provide similar acid

transport and reaction phenomena inside the fracture, and pressure inside conductivity cell is high enough to keep carbonate dioxide in solution. To achieve this result, the lab facility is to be customized for each stage of experiment. The experiment parameters, such as flow rate, flow pressure and reaction temperature, are studied carefully and set optimally. Then equipment's requirements can be specified. The goal is to assemble the equipment in a timely and cost effective manner, and make the labs easy to operate.

The previous studies conducted in acid fracturing conductivity were based on limited experimental conditions with limited types of formation rocks, and more important, the experiments did not represent typical field acid fracturing treatment conditions. The new lab facility is capable of conducting acid fracturing experiments at the conditions that are close to the field treatment conditions.

The new lab facilities are customized for the tasks. The fracture conductivity cell is per API standard and is modified to accommodate thick rock samples. The thick rock samples can create similar downhole leakoff velocity when acid flow into the fracture surface. The Chem/Meter pump can provide a pump rate that matches field operational conditions. All experimental variables are measured and recorded. The experimental data are processed and interpreted.

The laboratory set-up has three sections: acid etching section; rock surface characterization section; and fracture conductivity measurement section.

One complete acid fracture conductivity experiment includes the following steps:

- Core sample preparation: rock cutting, adding silicone rubber on sample sides, core sample saturation with water
- Initial core sample surface profile measurement and rock embedment strength test
- Acid preparation and injection through core samples
- Core sample acid etched surface profile measurement
- Measurement of core samples' weight and embedment strength after acid treatment
- Measurement of acid etched core sample fracture conductivity

Detailed procedures are developed for each experiment stage; the equipments and instruments specifications are listed; and the major lab setups are illustrated. Some preliminary results of the acid fracturing experiments are discussed.

CHAPTER II

ACID FRACTURE CONDUCTIVITY LAB SETUP

The acid fracture conductivity experiments are divided into three sections: acid etching process, core sample surface profile characterization, and acid fracture conductivity measurement section.

2.1 Acid etching treatment experiment

The acid etching experiment is to simulate field acid reaction with downhole carbonate formation rocks in an acid fracturing treatment. As illustrated in Figure 2.1, the acid fracturing experimental apparatus include the acid and brine storage tanks, high pressure Chem/Meter pump, 3/4" tubing and 1/2" hastelloy tubes, cylindrical heaters, API modified conductivity cell, back pressure regulators, pressure transmitters, Modbus data acquisition unit and various ball valves. In the experiment, acid is mixed in acid tank per each acid type recipe, and then is pumped and heated up and flows through the artificial fracture between the two core samples in the conductivity cell, and the fluids are collected in the spent acid tank.

During acid etching experiments, acid flowrate, acid temperature, pressure inside conductivity cell, leakoff differential pressure, and leakoff fluid volume are recorded. Pressures are controllable with back pressure regulators, which are using nitrogen. The high pressure pump discharges fluid at 1 L/min at 1000 psi. The heater can bring the fluid up to 300 F. The cell pressure is controlled with effluent back pressure regulator and the leakoff differential pressure is controlled by leakoff back pressure regulator. Spent acid is neutralized with caustic soda later. All the equipments reside inside laboratory exhaust system to vent the acid fume.

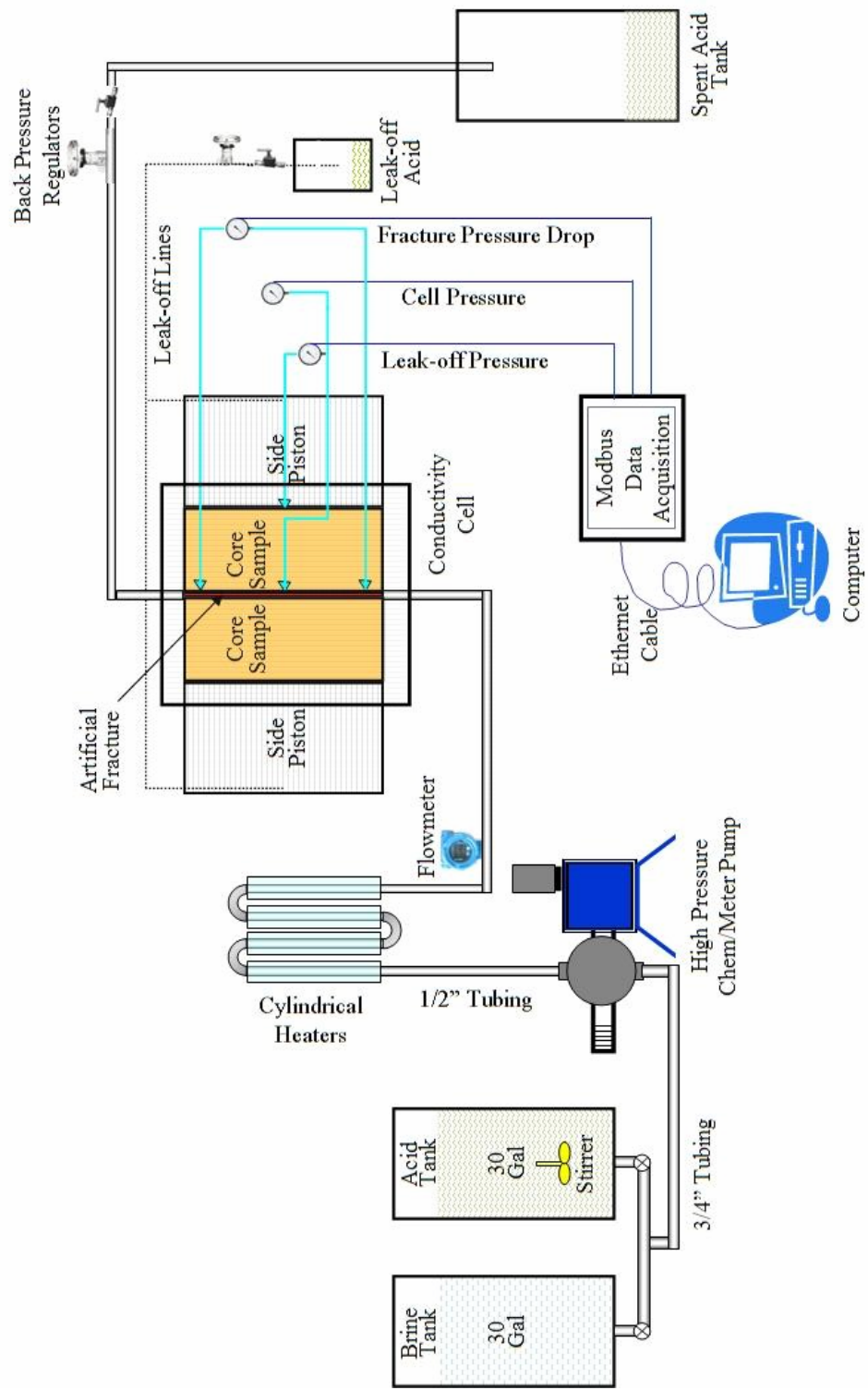


Figure 2.1 Acid fracturing treatment laboratory set-up

2.2 Core sample preparation

Core samples that used in acid frac experiments are: Indiana Limestone, Dolomite, Austin Chalk, and some carbonate cores from the fields. To better understand the relationship between rock type and acid etching, rock properties are measured including bulk permeability, porosity, and rock embedment strength. The core samples are cut to three inches thickness to fit in the customized conductivity cell. The shape and dimensions are shown in Figures 2.2, 2.3. For operational purpose, core samples are potted in high temperature RTV silicone rubber to provide a seal between the cores and the walls of the conductivity cell.

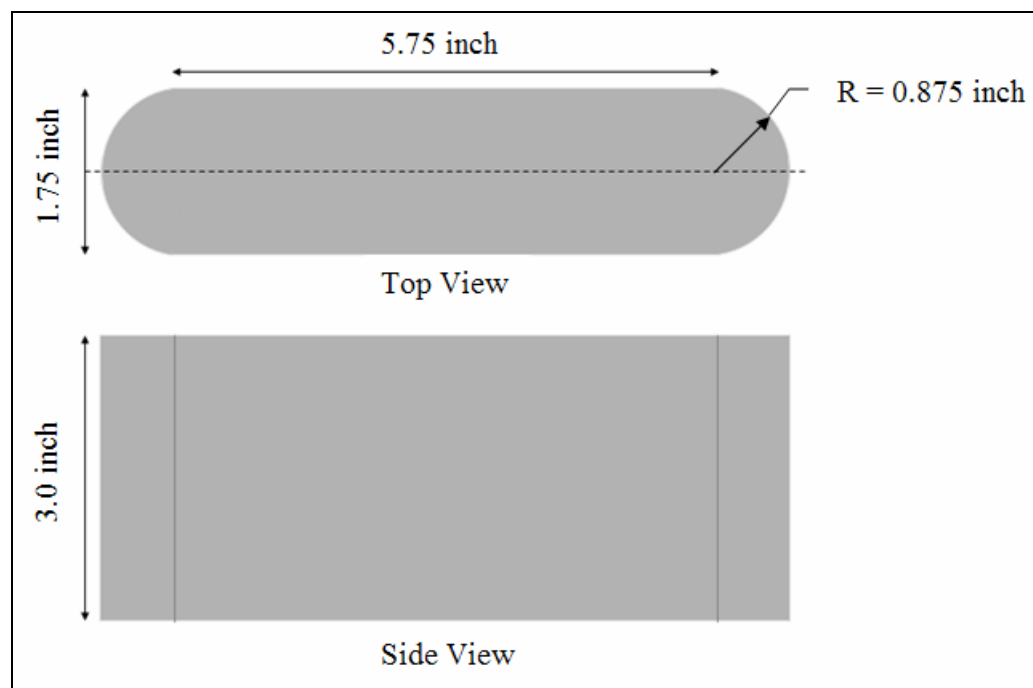


Figure 2.2 Dimensions of core samples



Figure 2.3 Core samples

2.3 API modified conductivity cell

The conductivity cell is designed per API standard, with modifications to ease the pressure measurement and fluid leakoff. Figure 2.4 shows the conductivity cell assembly and Figures 2.5, 2.6 and 2.7 show its components. Figure 2.5 shows the conductivity cell body. Dimensions of the cell body is 10" x 3-1/4" x 8", with a 7-1/4" x 1-3/4" hole through. For the 8 inch thick cell body, core sample thickness can vary from one inch to three inches. In acid frac experiments, most of the cores are cut to three inches thick and some of them are cut to two and a half inch, due to the source rock limitation. The advantage of the three inches thickness is that it helps monitoring the leakoff and wormhole phenomenon during acid injection. If the core sample is thin, acid can easily break through it with wormholes and it is difficult to control leakoff rate during experiments.

The side pistons, shown in Figure 2.6, are used to confine cores in the cell center and maintain a desired pressure inside cell body during acid treatments. Viton O-rings 2-350 VT90 are installed in the o-ring grooves and fit tight inside the cell. The piston's side to the core has a set of flow lines engraved. This design allows leakoff fluid flow out of cell body without additional pressure drop. There are also two flow inserts on

both ends of the cell body. They are the inlet and outlet for the flow through conductivity cell. Both flow inserts fit the cell body tightly and are sealed with Viton O-rings 2-123 VT90, as shown in Figure 2.7.

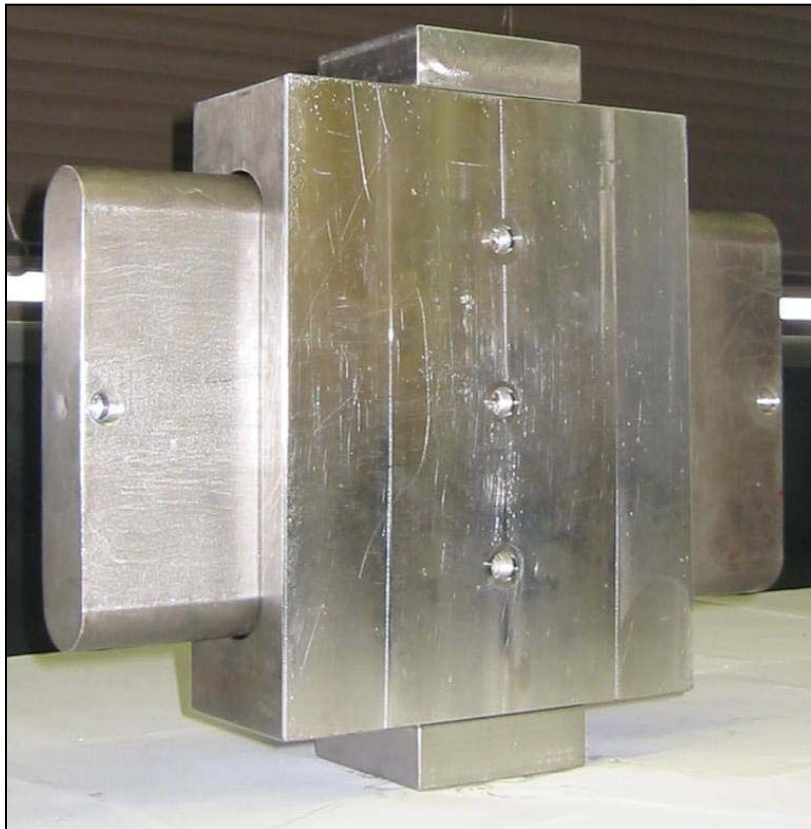


Figure 2.4 Fracture conductivity cell assembly with cores inside



Figure 2.5 Conductivity cell body



Figure 2.6 Side pistons

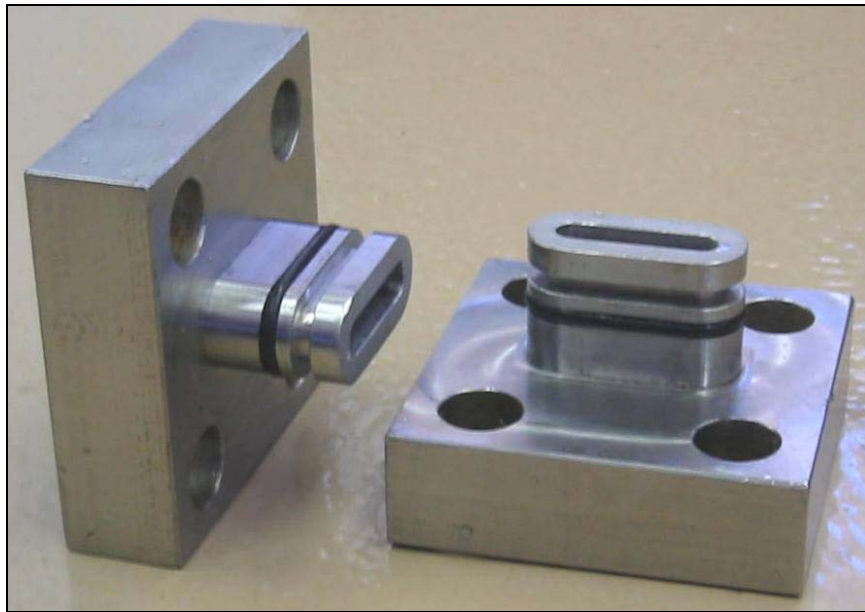


Figure 2.7 Flow inserts

The conductivity cell is made of hastelloy C276 material, which is acid resistant. There are three pressure access ports at one side of the cell body, as shown in Figure 2.4. The central one is for measuring the gauge pressure in the middle of the fracture and the two at the sides are for measuring the pressure drop along the fracture. Also, there is one port in each side piston, as shown in Figure 2.5. It is the flow path for leakoff fluid, and measuring the differential pressure across the core sample while leaking off.

The three ports on the cell body have 7/16" -20 SAE/MS female threads. The ports on the side pistons and the ports on flow inserts are of 1/4" NPT female thread. The flow inserts are mounted on the cell body with four pieces of 5/8" -1 inch long socket head screws each end.

The total weight of the conductivity cell assembly is about 110 Lbs. The acid is injected into the conductivity cell at about 1000 psi, so that all CO₂ gas generated during acid carbonate reaction will be kept in solution and does not aerate to form bubbles. Otherwise bubbles inside the fracture will divert acid flow and force the fluid to leak off.

Then additional worm holes may be present, which is not the case during field acid frac treatment.

The side piston cross-section area is about 12.47 inch². 1000 psi pressure inside conductivity cell body generates about 24,940 Lbs force against the side pistons. To keep the side pistons in position during acid injection, a hydraulic jack and two screw jacks are installed to accommodate the conductivity cell assembly. As shown in Figure 2.8, looking from the above, the horizontal screw jacks confine the whole assembly in place throughout the experiment. The conductivity cell is placed in such a position that the fluid will flow vertically from bottom up to top. Since the fluid flows vertically, there will be little gravity effect on the fluid concentration distribution and eliminate the difference between the two core samples.

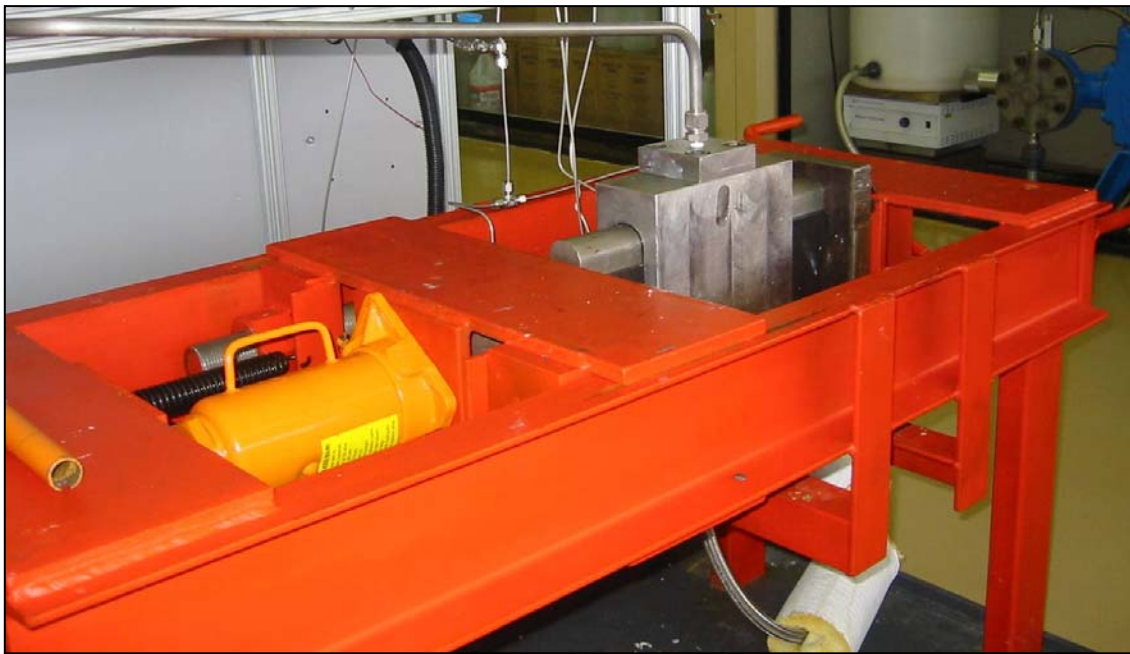


Figure 2.8 Modified hydraulic jack with conductivity cell

The same conductivity cell is used for fracture conductivity measurement. It is carried out in a heavy duty load frame. The conductivity cell is supported by a rack on the load frame to properly space out the core sample in the cell. A proper rack height

allows the flow right through the interface between two cores. Figure 2.9 shows the positions of the support rack and the cell body inside the load frame.

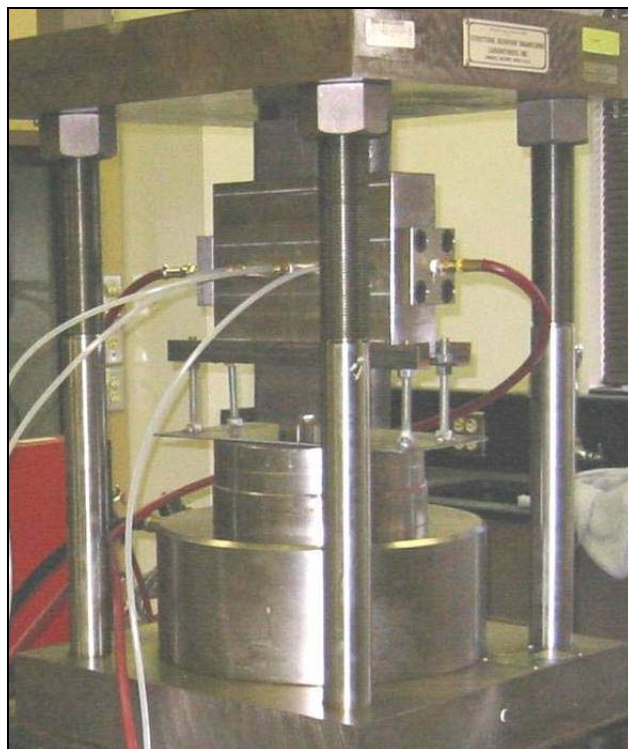


Figure 2.9 Conductivity cell and support rack inside load frame

2.4 Acid pump

Real acid fracturing jobs are pumped at high rate to stimulate well production. To mimic the field job conditions, the lab experiments require flow rate 1 L/min at about 1000 psi. We searched the available pumps in the market and found Chem/Meter 802 pump fit our application requirement.

The pump is a hydraulic displacement pump and uses a hydraulically balanced diaphragm to pump the process fluid. Its maximum operating pressure is about 2200 psig and max flow rate is about 1.4 L/min. There is an externally adjustable relief valve in the hydraulic system to protect the pump against excessive pressure. The pump rate can be manually regulated from 0 to 100% of maximum rated capacity while stationary

in operation. All the flow-wet components are made of acid corrosion resistant material. The inlet and outlet check valves allows single direction flow. It is crucial to check the direction when reassemble them. Figure 2.10 displays the pump set up in the lab. Figure 2.11 is Chem/Meter pump calibration chart. The pump rate increase from 0 to about 1.4 L/min while changing control rod from 0% to 100%. Note that it is not a linear relationship.



Figure 2.10 High pressure Chem/Meter pump

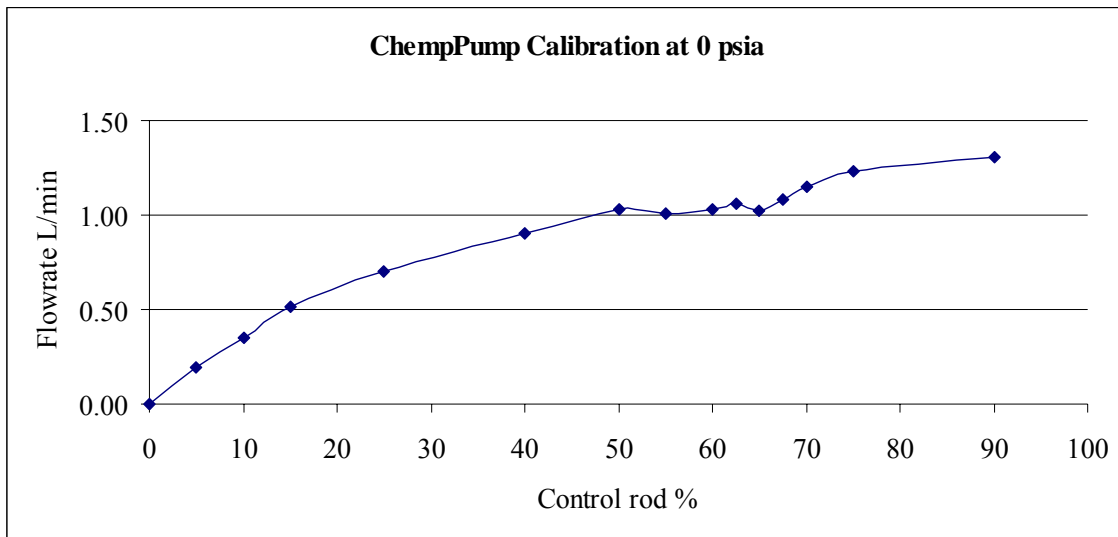


Figure 2.11 Chem/Meter pump calibration chart

At the pump suction end the check valve has a 3/4" NPT male thread. It is connected to acid tank and water tank via 3/4" ID braided PVC hoses and adapters. The Nalgene tanks have 30 gallons capacity and are made of high density polyethylene. Acid and water tanks are separated by 3/4" PVC ball valves. All the materials are corrosion resistant.

At the pump discharge end the check valve has another 3/4" NPT male thread. It is connected to 1/2" OD x 0.402" ID hastelloy tube via a 3/4" NPT female x 1/2" Gyrolok compression fitting. All the tubes and connectors from the pump discharge end are rated to at least 3,000 psi working pressure.

2.5 Data acquisition

The acid frac experiment variables, including acid flow rate, acid temperature, pressure in the middle of the conductivity cell, pressure drop along the fracture, the leakoff differential pressure, are digitalized with sensors and recorded in database.

The acid flowmeter is Sponsler IT400 model. It runs on battery. The LCD display has real time flow rate indication and also totalization volume indication. The

flowmeter has 1/2" NPT male threads on both ends. It requires about 12 inch straight line before fluid flow through it for accurate rate reading.

The pressure inside the fracture cell is detected with Honeywell gauge pressure transmitter. Its work range is from -15 psi to 3000 psi. The pressure drop along the fracture and the leakoff differential pressure is measured with Honeywell smart differential pressure transmitters. Its work range is from -5 psi to 100 psi. The pressure transmitters display pressure data on a LCD screen and output 4~20 mA DC current signals. These pressure transmitters are connected to Acromag Modbus TCP/IP Ethernet I/O modules via grade 16AWG electric cable. The power supply for the modbus module and pressure transmitters is a 30 watt 18V single DC output adapter. The pressure transmitters are connected to the ports on the conductivity cell assembly with 1/8" hastelly C276 tubes and Gyrolok compression fittings. Figure 2.12 illustrates the pressure transmitters' setup.

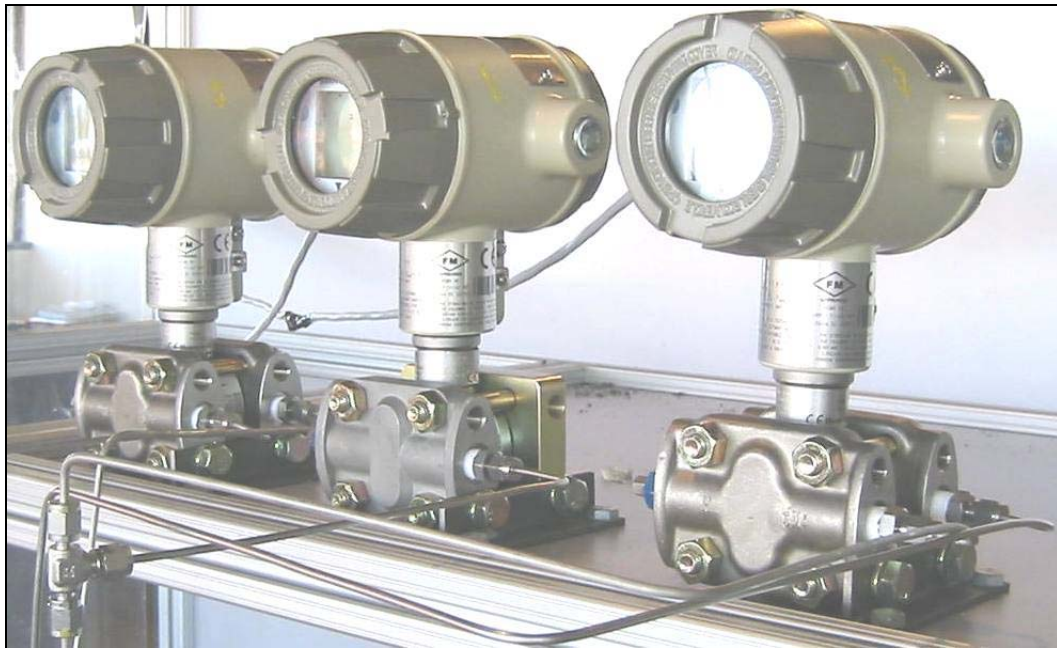


Figure 2.12 Honeywell pressure transducers

Acromag modbus TCP/IP module is utilized to transfer the signals to computer. It has a direct network interface, processes I/O signals on up to twelve channels, and handles power conversion. The I/O modules are configured using internet explorer, as shown in Figures 2.13, 2.14, 2.15. The web pages guide user through the steps to configure network settings, calibrate module and test operation. The modbus is connected to computer with a standard CAT5 network crossover cable. The modbus module assumes a static IP address “128.1.1.100” and a default subnet mask of “255.255.255.0”. The computer is the server with IP address “128.1.1.25” and opens port number 502 for modbus module.

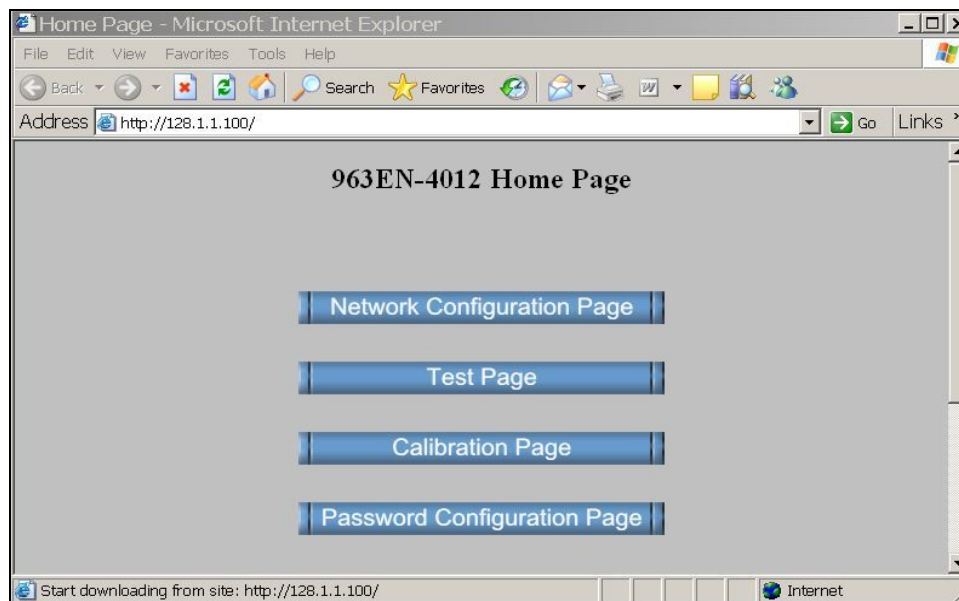


Figure 2.13 Modbus TCP/IP web browser configuration pages (1)

963EN-4012 Network Configuration Page

Static IP Address.....128.1.1.100 Number of Sockets.....10

DNS Server.....128.1.1.10 Subnet Mask.....255.255.255.0

Gateway.....0.0.0.0 Host Name.....963en

Active IP Address.....128.1.1.100 MAC Address....00:01:C3:00:05:C3

☒ Use Static IP Addressing
☐ Use DHCP/BOOTP
☐ Use DHCP/BOOTP W/Fallbacks

submit

Wink On/Off

Figure 2.14 Modbus TCP/IP web browser configuration pages (2)

963EN-4012 Test Page

Input Values

Port 0		Port 1		Port 2	
CH 0	3.994 mA	CH 4	0.000 mA	CH 8	0.000 mA
CH 1	3.965 mA	CH 5	0.000 mA	CH 9	0.000 mA
CH 2	3.937 mA	CH 6	0.000 mA	CH 10	0.000 mA
CH 3	0.000 mA	CH 7	0.000 mA	CH 11	0.000 mA

Port Configuration Control

Port	Range	Port	Range	Port	Range
0	<input checked="" type="radio"/> 0 - 20 mA	1	<input checked="" type="radio"/> 0 - 20 mA	2	<input checked="" type="radio"/> 0 - 20 mA
	<input type="radio"/> 4 - 20 mA		<input type="radio"/> 4 - 20 mA		<input type="radio"/> 4 - 20 mA
	<input type="radio"/> 0 - 11.17 mA		<input type="radio"/> 0 - 11.17 mA		<input type="radio"/> 0 - 11.17 mA
	<input type="radio"/> 0 - 1 mA		<input type="radio"/> 0 - 1 mA		<input type="radio"/> 0 - 1 mA

submit

Figure 2.15 Modbus TCP/IP web browser configuration pages (3)

The modbus receives the DC current signals and stored the data in its registers. There are 4 types of registers and they have different addresses. In our application, the data for each channel are in input registers with addresses 30017 for channel 0, 30018 for channel 1 and 30019 for channel 2. Data acquisition in computer is programmed with LabVIEW software. As shown in figures 2.16, 2.17, the program reads the registers and displays the data on a wave chart. At the same time the readings are written to an excel file in the same time sequence. The program is quite flexible to read up to 12 channels from modbus module. The DC current value is then processed in excel spread sheet to represent the exact pressure values.

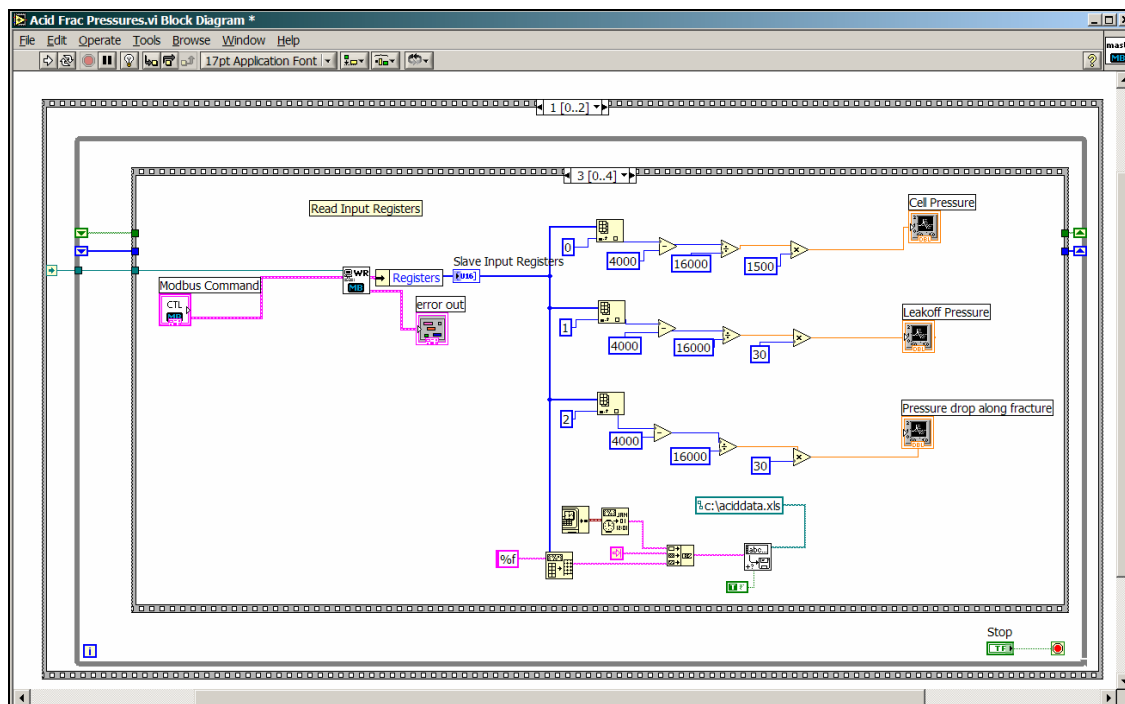


Figure 2.16 Data acquisition LabVIEW program (block diagram)

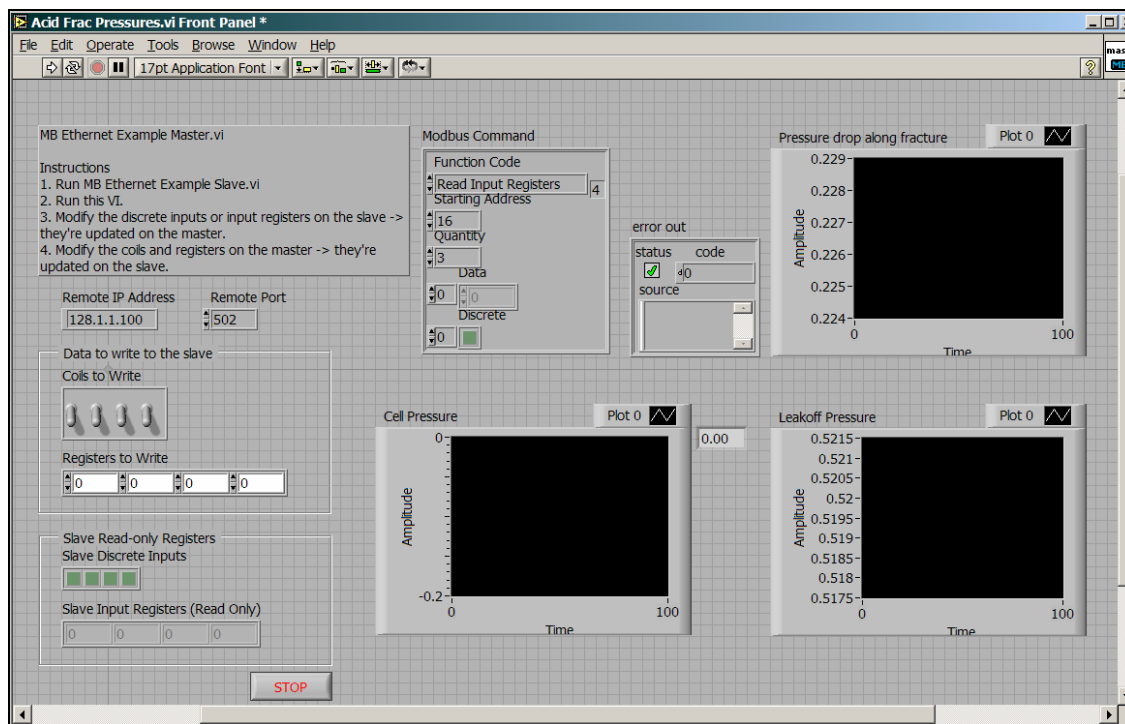


Figure 2.17 Data acquisition LabVIEW program (front panel)

2.6 Cylindrical heaters

In most field acid fracturing treatments, the acid is pumped into and reacts with reservoir rocks at a temperature that is much higher than the atmospheric temperature. From chemical reaction kinetics study, we know that the reaction rate of hydrochloric acid and carbonate changes drastically with temperature changes. To better represent the acid reaction with the downhole formation rocks, the acid is heated up before it flows through the cores. The Omegalux CRWS series semi-cylindrical ceramic radiant heaters are used in the lab to heat up the fluid during pumping. The heaters are produced using high purity vacuum formed ceramic fiber, with low sodium inorganic bond. The heating elements are helically wound iron-chrome-aluminum wires which are imbedded into the vacuum formed ceramic fiber.

The heater assembly is hollow through the center, leaving 1.5 inch diameter holes at both ends. Each semi-cylindrical radiant heater is 1200 watt. Eight 27-inch-

long cylinders are used, based on the experiments requirement of 200 degree F. The flow lines go through the center of the cylindrical heaters. They are connected to 240V AC power supply and controlled with thermal couple to maintain a constant temperature during each experiment. Figure 2.18 shows the heaters connection and set up in the lab cart. The thermal couple sensor is located about 5 inch downstream the flow tube and the temperature is displayed in the screen. The switch for the heater is close to the conductivity cell assembly. It is important not to leave the heaters on for a long time when there is no fluid flow in the flow line, as the heaters temperature can increase to over 1000 °F and degrade the metal tube. From our experience, it will help to increase fluid temperature faster if switch on the heater 5~10 minutes before turning the pump on.



Figure 2.18 Omegalux cylindrical heater

2.7 Back pressure regulators

As discussed in previous section 2.3, the pressure inside the conductivity cell is required to be about 1000 psi during acid injection. The pressure is achieved with a back pressure regulator in the flow line.

Tescom back pressure regulator model 26-1755 is installed on the conductivity cell effluent flow tube. This regulator is designed for high pressure applications. It has a metal to metal soft seated design and is dome loaded. The ratio of flow line pressure over nitrogen dome pressure is about 16, which means, that the nitrogen dome pressure should be about 62 psi to achieve 1000 psi pressure in the flow line. To resist acid corrosion, its flow wet components are made of hastelloy C material. The fluid inlet and outlet ports are both 1/4" NPT female threads. The nitrogen input port is on top of the dome and it has a 1/8" NPT female thread.

Mity-mite back pressure regulator model S91-W is installed on the leakoff line. Its nitrogen input pressure range is 100 ~ 2000 psi. The pressure in the leakoff line is the same as the nitrogen dome pressure. The connections to the flow lines are 1/4" NPT female threads and the connection to nitrogen line is 1/8" NPT female thread. A gas pressure gauge is mounted on the top of the back pressure regulator to monitor the nitrogen dome pressure. There is a ball valve installed in the leakoff line in front of the regulator so that the leakoff line can be shut off if no leakoff is required during pumping.

Both back pressure regulators are operated independently. Their dome pressures are supplied by different nitrogen bottles with different scale regulators. Note that Tescom BPR's ratio of flowline pressure over gas dome pressure is about 16 and Mity-mite's is 1. It is important to make sure that the domes are pressurized before turning on the pump. Also, never over charge Tescom regulator's dome pressure. To better control the leakoff rate it is advised that always has a higher initial leakoff line dome pressure than the main flow line. This also helps to prevent bursting the Teflon diaphragm in the mity-mite regulator. Figures 2.19 and 2.20 show both regulators set up on the lab cart.



Figure 2.19 Tescom back pressure regulator



Figure 2.20 Mity-mite model S91-W back pressure regulator

2.8 Vacuum saturation facility

The vacuum system is used to completely remove air in core sample's pore space and then saturate the cores with water. As shown in Figure 2.21, the setup includes a vacuum pump, a large size glass vessel and a water container. Normally it takes about one hour to vacuum the core samples and saturate them with water.



Figure 2.21 Vacuum vessel with cores immersed in the water

2.9 Rock surface profile measurement setup

A profilometer was designed to measure the rock's surface profile before and after acid reaction. The system composes an automated XY coordinate's movement system and Acuity AR200 laser measurement sensors. The laser sensors project a beam of visible laser light that creates a spot on the core surface. Reflected light from the

surface is viewed from an angle by a line scan camera inside the AR200 sensor. The core's distance is computed from the image pixel data. The mechanical movement system moves the laser sensor in the X and Y directions covering the whole core surface area, while LabVIEW program recording the height in the Z direction at the designated pace. The system also uses LabVIEW program to control the automated measurement and plot the core surface profile in 3-D graph.

Figures 2.22, 2.23 show the profilometer setup and the sample plot it generated. More details about the profilometer will be discussed in a separate thesis (Camilo, 2006).

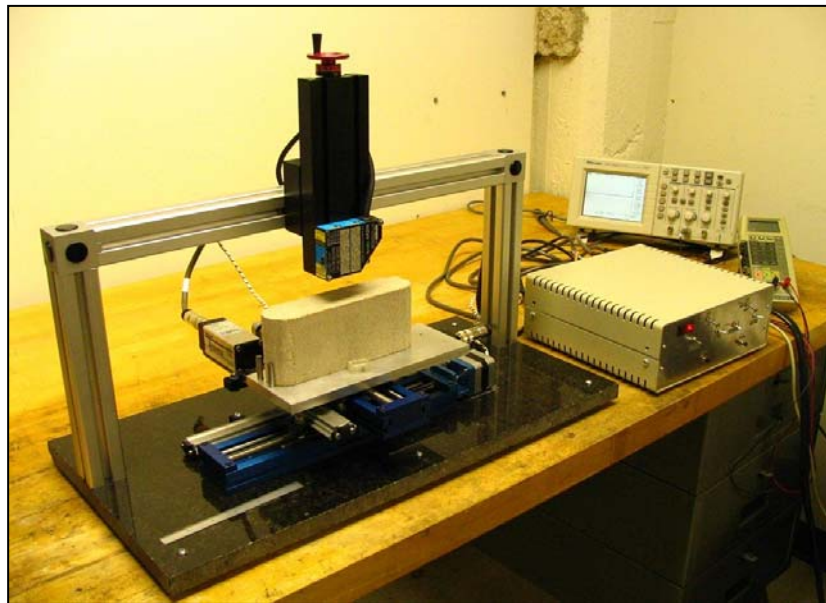


Figure 2.22 Profilometer

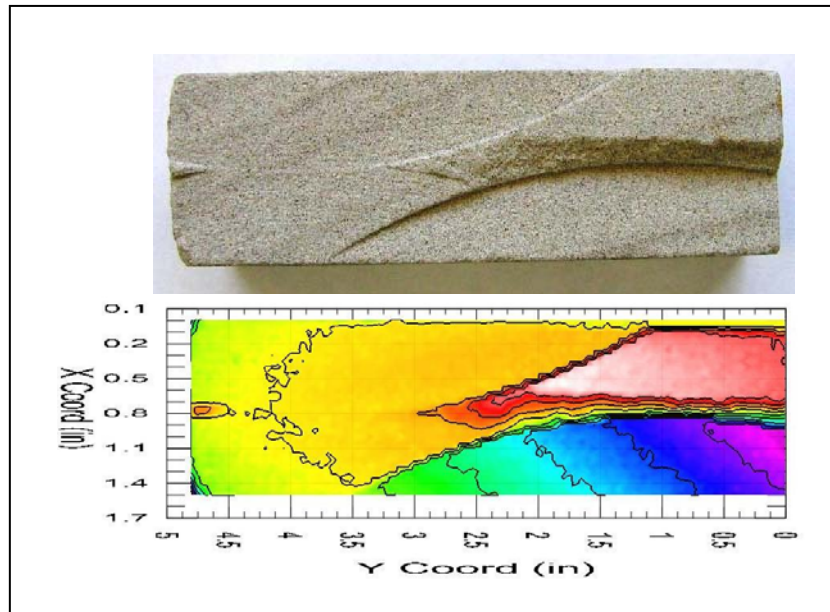


Figure 2.23 Rock profile plot sample

2.10 Fracture conductivity measurement experiment setup

The fracture conductivity measurement experiment is to quantify the acid etching effects. We measure the fracture conductivity by flowing nitrogen through a pair of acid etched core samples, and calculating the fracture permeability with Forchheimer's equation. This method was developed by Pursell⁶ in 1987. Figure 2.24 illustrates conductivity measurement process. The major components are nitrogen supply, nitrogen mass flow controller, acid etched core samples, conductivity cell, load frame, pressure transmitters, and back pressure regulator.

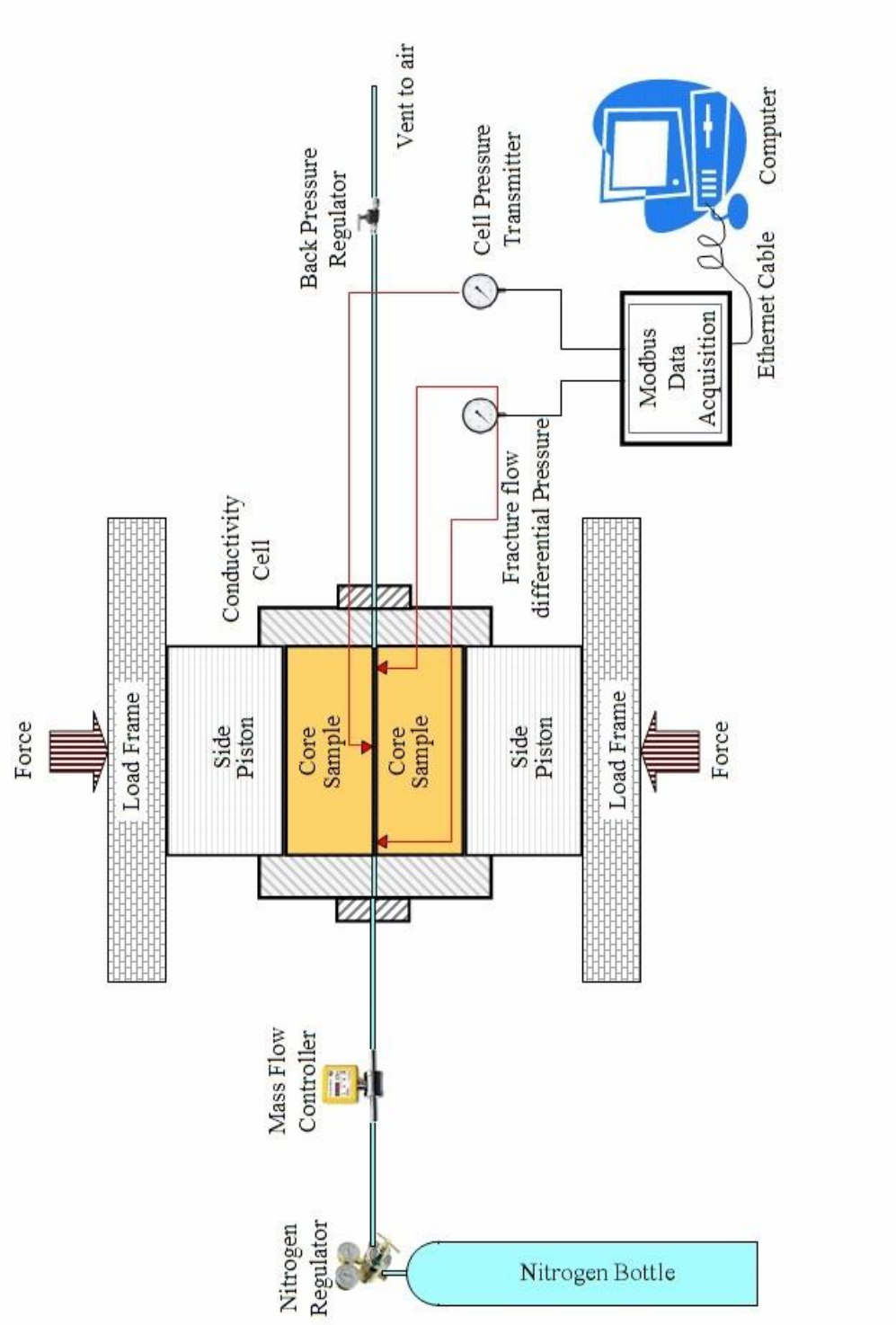


Figure 2.24 Fracture conductivity measurement illustration

The fracture conductivity is measured under step-changed closure stresses, simulating field downhole fracture closure pressure after pumping stops. In the lab, closure stress is produced with the load frame CT-250, manufactured by Structure Behavior Engineering Laboratories, Inc. This compression tester has a ram area of 125 square inches requiring 2,000 psi to produce 250,000 lbf force, the capacity of the loading frame. An AP-1000 pump system is used to pressurize hydraulic oil for the load frame. The pump is operated by compressed air. Supplying 100 psi of compressed air to the pump produces maximum 1,000 psi discharge pressure for the hydraulic oil. When the maximum pressure is applied to the loading frame ram (125 sq. in.), 125,000 lbf of axial force is produced. Then this amount of force is transmitted to the core samples, which generates about 10,000 psi compression stress on the cores interface, since the core sample cross-section area is about 12.47 in². The hydraulic oil discharge pressure is controllable. In the experiments, the closure stress is increased gradually. The fracture conductivity is measured under 100 psi, 500 psi, 1000 psi, 2000 psi, 3000 psi, 4000 psi, 5000 psi and 6,000 psi closure stresses.

Figure 2.25 shows the actual fracture conductivity measurement lab setup: load frame, conductivity cell and others key components pictures. The nitrogen flow rate is measured and adjusted with Aalborg GFC Mass Flow Controller model 47. The stream of nitrogen entering the mass flow transducer is split by shunting a small portion of the flow through a capillary sensor tube. The remainder of the gas flows through the primary flow conduit. In order to sense the flow in the sensor tube, heat flux is introduced at two sections of the sensor tube by means of precision wound heater-sensor coils. Heat is transferred through the thin wall of the sensor tube to the gas flowing inside. An output signal is generated that is a function of the amount of heat carried by the gases to indicate mass-molecular based flow rates. This mass flow controller incorporates a proportionate solenoid valve, which can correct flowrate deviation from the set point by compensating valve adjustments and thus maintain desired flow parameters. Note that 15 minutes warm-up period is required before flow any gas through the flow controller. To achieve desired flow rate, use the built-in set point

potentiometer located near the solenoid valve. While applying flow to the transducer, adjust the set point with an insulated screwdriver until the flow reading is the same as the desired control point. The flow rate is displayed in liter/min in the LCD screen.

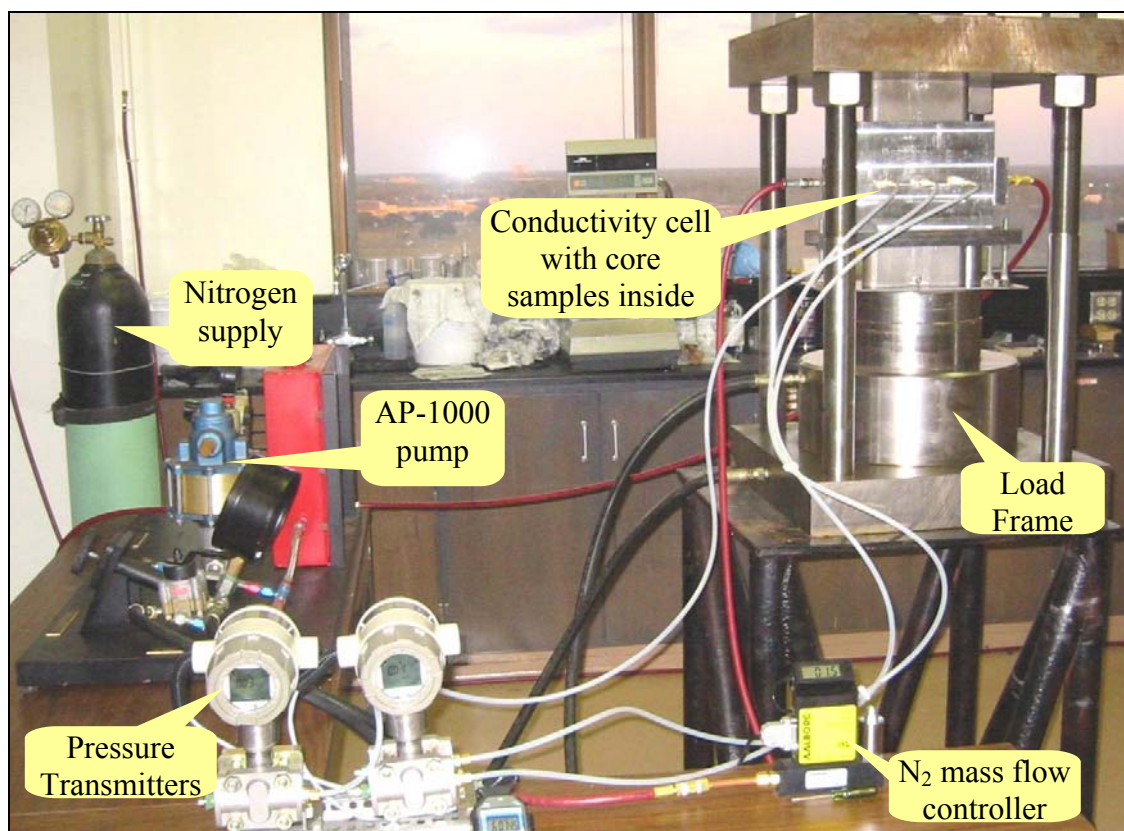


Figure 2.25 Load frame and others key components

Under each specific fracture closure stress, four different flow rates are tried. To apply Forchheimer equation properly, the conductivity cell pressure has to be constant during all four flow rates. This is achieved with an APCO back pressure regulator (BPR). The BPR is installed on the nitrogen effluent line from the conductivity cell. Normally the conductivity cell pressure is set at 50 psi. It is measured with a Honeywell gauge pressure transmitter. Another Honeywell differential pressure transmitter is used to measure the nitrogen pressure drop along the fracture. The nitrogen temperature is

detected also. All the experimental variables are recorded in Excel spreadsheet and processed to draw Forchheimer's charts. Fracture conductivity is read from the chart then. Figure 2.26 is a sample chart.

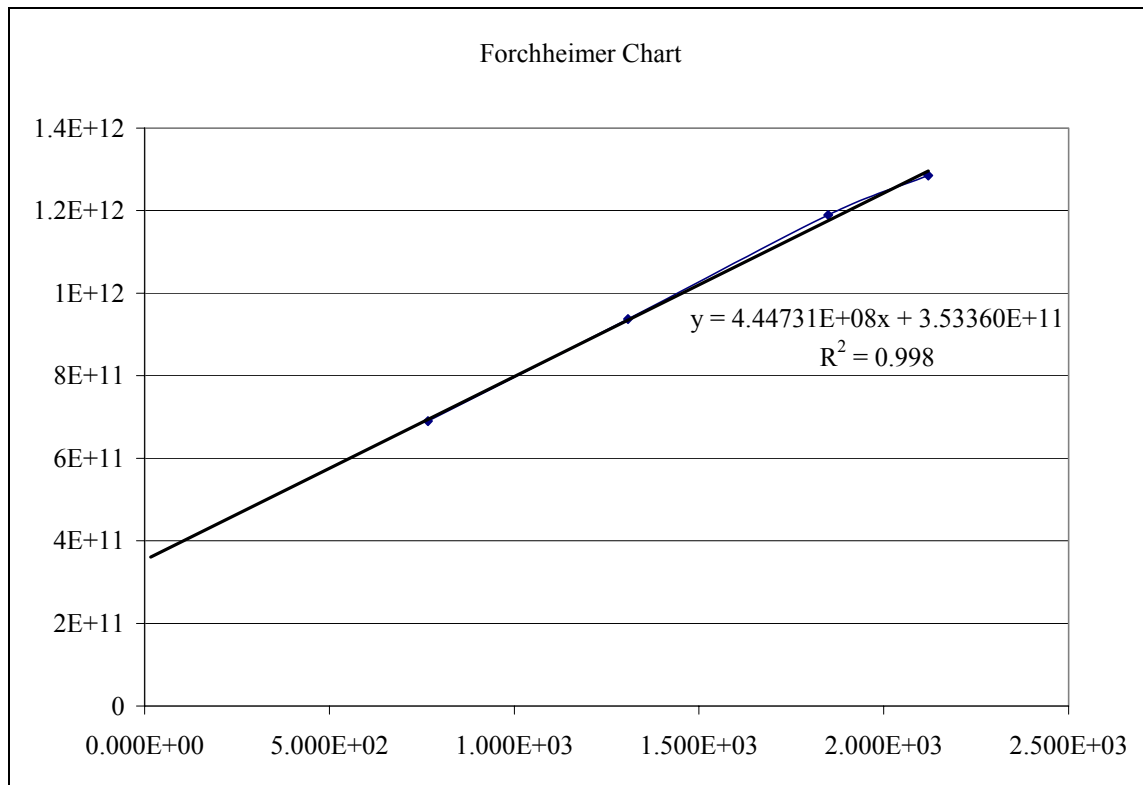


Figure 2.26 Sample Forchheimer chart

CHAPTER III

EXPERIMENTAL STUDY OF ACID FRACTURE CONDUCTIVITY

3.1 Acid fracturing experimental parameter study

A major objective of the research project is to scale up the experimental conditions that are as close to the actual field acid fracture treatment conditions as possible. We try to flow the acid through the fracture at a realistic rate which represents acid flux in a field job and maintain the conductivity cell temperature similar to reservoir temperature. Thus the experimental results will be representative and can be used to guide field acid fracture job design with more confidence.

In the field acid fracturing treatments, the acid flow rate ranges from 10 BPM to 50 BPM. The flow rates vary significantly because of different target zones depth, treatment intervals length and field location logistics. The reported fracture width is about 0.1 to 0.2 inch during acid injection and the fracture height ranges from 50 ft to 100 ft.

Carbonate rock reacts with hydrochloride acid fast at the fracture surface⁸. The control factor in acid fracturing process is the mass transfer rate. To match the field treatment conditions, we try to have the same Reynolds number in the lab experiments as that in field acid injections. Reynolds number is ratio of convective transport to viscous resistance. It is proportional to the ratio of inertial force to viscous force, and is used in momentum, heat and mass transfer to account for dynamic similarity. If Reynolds numbers are the same, the acid mass transfer rate in the lab is comparable to that of field acid fracturing jobs.

Assume the acid fluid viscosity is constantly 1 cp, the flow is steady and the liquid fluid is incompressible, the initial acid flow Reynolds number is calculated as:

$$N_{Re} = \frac{Dv\rho}{\mu}(\text{pipeflow}) = \frac{w_f v \rho}{\mu}(\text{fractureflow}) \quad (3-1)$$

For a field fracturing job, there are two symmetrical fractures. So, pumping rate:

$$q = vw_f \times 2h \quad (3-2)$$

Replace wfv with q and h , then

$$N_{Re} = \frac{q\rho}{2h\mu} (field, 2 wings) \quad (3-3)$$

Where q is acid injection rate, μ is acid fluid viscosity, h is fracture height, ρ is acid fluid density, w_f is fracture width, and v is acid flow velocity.

For acid leakoff control in lab experiments, we use the Peclet number to match field condition. The Peclet number is ratio of convective transport to diffusive transport. Used in mass transfer in general and forced convection calculations in particular, it is defined as:

$$N_{Pe} = \frac{vD}{\alpha} (cylindrical) \quad (3-4)$$

In a fracture flow, the leakoff Peclet number is defined as

$$N_{Pe} = \frac{\bar{u}_y w_f}{2D_{eff}} \quad (3-5)$$

Since

$$q = \bar{u}_y \times 2h \times 2x_f = 4\bar{u}_y h x_f, \quad (3-6)$$

Then

$$N_{Pe} = \frac{qw_f}{8hx_f D_{eff}} \quad (3-7)$$

Where q is the injection rate, D_{eff} is the effective diffusion coefficient, h is the fracture height, w_f is the fracture width, u_y is the fluid diffusion velocity, and x_f is the fracture length.

Table 3.1 gives the summary of a typical field acid fracturing job. The acid flow rate, formation height, fracture width and length, fluid viscosity and density are listed.

Table 3.1 Data of a typical field acid fracturing treatment

Acid pumping rate, q	20 <i>bbl/min</i>	0.053 m^3 / sec
Formation height, h	100 <i>ft</i>	30.5 <i>m</i>
Fracture width, w_f	0.2 <i>inch</i>	0.0051 <i>m</i>
Fracture length, x_f	100 <i>ft</i>	30.5 <i>m</i>
Fluid viscosity, μ	1 <i>cp</i>	0.001 $N - S / m^2$
Fluid density, ρ	1000 kg / m^3	1000 kg / m^3

$$N_{Re} = \frac{q\rho}{2h\mu} = 869 \quad (3-8)$$

$$D_{eff} = 8.8 \times 10^5 \text{ cm}^2 / \text{sec}. \quad (3-9)$$

$$N_{Pe} = \frac{qw_f}{8hx_f D_{eff}} \quad (3-10)$$

Assuming that the fracture width is set at 0.125 in, with a cell width of 1.75inch (0.044m) in the lab experiments, the lab injection rate should be:

$$q = N_{Re} \times \frac{h\mu}{\rho} = 869 \times \frac{0.044 \times 0.001}{1000} = 3.82 \times 10^{-5} m^3 / \text{sec} = 2.29 \text{ L} / \text{min}. \quad (3-11)$$

From the calculation, the flow rate is required to be 2.29 L/min in the lab, regardless the fracture width in the conductivity cell, as far as it is relatively small comparing to the cell width. However, no high-pressure high-rate pump in the market meets this requirement. The best fit is the Chem/Meter pump model 802, which can pump at about 1 L/min at 2,200psi discharge pressure. This rate is much lower than the calculated 4.6 L/min rate.

3.2 General experimental procedure

3.2.1 Acid etching procedure

- Prepare core samples;
- Saturate core samples with brine;
- Prepare acid and brine;
- Assemble core samples into conductivity cell and set at desired fracture width;
- Pump heated acid in between rock samples to etch the fracture surfaces;
- Switch the pump suction to water tank and flush the rock samples;
- Disassemble the unit and clean up.

3.2.2 Fracture conductivity measurement procedure

- Prepare and load acid etched core samples into conductivity cell
- Place conductivity cell in load frame and connect flow lines
- Apply desired overburden pressure on core samples and let stabilize
- Flow nitrogen and record data at different flow rate
- Increase overburden pressure and repeat last two steps
- Process data and calculate the conductivity

3.3 Core preparation procedure

The cores are precisely cut the same shape as the hole inside the conductivity cell and are 0.07 inch less in size on each side. The core sample is then potted in high temperature RTV silicone rubber. There is a mold of the same dimensions as the hole in conductivity cell. The core sample is placed in the mold, and then the liquid silicone is injected into the gap between the mold and the core. The liquid silicone will turn into solid rubber when put in the oven at 200°F for one hour. The solid silicone rubber helps to provide sealing between the core and the conductivity cell during acid etching and conductivity measurement experiments. Next the core sample weight and thickness is measured and recorded. These data are compared with the weight and thickness data

after acid etching, so that we know how much carbonate has reacted with acid and the change of core samples thickness.

3.4 Core sample saturation procedure

All the core samples are vacuumed and then saturated with water before acid etching treatment. Otherwise the pore space in core samples may trap active acid and cause excessive wormholes effect, which does not represent typical downhole conditions.

The core sample saturation is a simple process. It is done in the department's existing vacuum system. The procedure is as following:

1. Connect the vacuum lines to the vacuum pump, glass vessel, buffer bottle and the switch.
2. Put special vacuum grease on glass vessel rim.
3. Put core samples in glass vessel, and then move glass lid to cover the vessel completely.
4. Turn on the vacuum pump and keep it running for 1 hour.
5. Close the valve between the vacuum pump and the glass vessel.
6. Connect the vacuumed glass vessel to the water source with the branch line. Water will be sucked into the core sample vessel. Make sure the whole core sample is submerged.
7. Shut off the vessel vacuum lines.

When ready to do experiment, take the core samples out of the vessel. Keep in mind to minimize the time exposing the saturated core samples in the air.

3.5 Acid etching treatment procedure

Acid etching treatment is the key process of an acid fracture conductivity experiment. It mimics the acid-carbonate reaction on the fracture surfaces at a desired temperature. A variety of acid fluids are used to etch carbonate core samples at three different contact times: 15 min, 30 min and 60 min.

During acid etching treatment, researchers deal with highly corrosive fluids at high temperature and high pressure. Safety is the first priority when conducting experiments. All safety gears, including masks, goggles, protective clothing and shoes, should be worn. Other safety measures, including acid spill kit, fire extinguisher, and MSDS documents for all chemicals, should be prepared at appropriate locations in the lab. The acid containers and experimental apparatus are located in a fume hood and a canopy hood separately, and the exhaust system should be kept on during the experiments. When all the necessary set up is finished, the experiment begins. The procedure is as:

1. Check the valves under the acid tank and the water tank in close position. Fill the water tank and the acid tank with tap water. The water volume for acid tank is calculated base on the treatment fluid recipe, the desired acid contact time, and the dead volume in the tank. The pumping rate is about 1 L/min.

2. Turn on the lab exhaust system. Add the corrosion inhibitor and other chemicals (if any, like gelling agent, emulsifier, etc.) in the acid tank. Lastly add concentrated hydrochloric acid (31.45% by weight) to the mixture. Adjust Barnstead maxi stirrer and let it run for 30 minutes to mix the fluid completely.

3. Assemble the conductivity cell: put Dow Corning grease around the core samples; identify the correct sides and insert them into the cell body; put the shims in the middle of the cell (the shims width is the fracture width); use the hydraulic jack push the core samples to center; use a syringe to inject sealant around the core samples; then use the hydraulic jack to push the side pistons to position; set the screw jack to confine the core samples and the side pistons in the cell; pull the shims out of the assembly and install the flow inserts.

4. Connect the flow tubes, the leakoff lines and the pressure access lines to the fracture conductivity cell. Then check the flow lines all the way from the water and acid tanks to the spent acid tank. Make sure all the connections are tightened and the valves are in proper positions. Also check the connections on the nitrogen lines from the nitrogen bottles to the back pressure regulators.

5. Set the heater controller: the upper range is 10°F higher than the desired acid temperature and the lower range is about 5°F higher than the desired temperature. For example, if the acid-rock reaction temperature is to be 200°F, the heater controller will set 210°F as upper limit and 205°F as lower limit. During a few tests, this setting ensures the fluid temperature about 200°F in the conductivity cell.

6. Fill the acid frac experiment data sheet. Run the LabVIEW program “acid frac pressure.vi” from the lab computer. The data and charts for the three pressure channels display on computer screen.

7. Open the nitrogen regulators. For the leakoff back pressure regulator, adjust its nitrogen outlet pressure to about 1000 psi. For the main effluent back pressure regulator, adjust its nitrogen outlet pressure to about 10 psi, which will apply a 150 psi back pressure in the main flow line.

8. Open the water tank valve and then switch on the Chem/meter pump. Observe the pressure transmitters and check the effluent. Check the connections and make sure no leakage.

9. Adjust the nitrogen regulator for the effluent back pressure regulator to increase the back pressure to 1000 psi. The pressure should be adjusted gradually to avoid any shock to the system. Check the flow line to make sure no leakage at high pressure.

10. Adjust the leak off back pressure with its nitrogen regulator to achieve an ideal leak off rate. Use some glassware to collect the leakoff fluid.

11. Monitor the fluid temperature constantly. When it reaches the desired reaction temperature, open the acid tank valve and close the water tank valve.

12. Pump the acid fluid and monitor the acid tank fluid level constantly. Switch the pump suction to water tank when finish acid pumping. Flush the conductivity cell assembly with water for 15 minutes. At the same time, change the leakoff acid collector to another empty glass to collect leakoff water. Increase the leakoff rate so that the core sample can be cleaned up quickly.

13. Switch off the Chem/Meter pump after water flush. Then close the nitrogen bottles and bleed off the dome pressures from the back pressure regulators. The acid etching treatment is complete.

14. Unload the hydraulic jack. Apply about 50 psi back pressure on the main effluent line. Switch on the Chem/Meter pump with water supply on. One side piston will be pushed out from the conductivity cell body.

15. Disconnect all connections from the conductivity cell. Use the hydraulic jack to push the other side piston and the core samples out of the conductivity cell body. Rinse all components with water.

16. Stop running the LabVIEW program. Rename the data file and process the pressure data in Excel spreadsheet and save the files.

Note: If observe any abnormal phenomenon, find the cause and make necessary adjustment or shut down the Chem/Meter pump immediately during experiments for any safety concern.

3.6 Fracture conductivity measurement

Fracture conductivity is defined as:

$$C_D = k_f w \quad (3-12)$$

Where k_f is fracture permeability and w is the fracture width.

The fracture conductivity measurement has been studied by many researchers. We use the method developed by Pursell⁶.

Forcheimer's equation is applied for high rate nitrogen flow in porous media:

$$\frac{p_1^2 - p_2^2}{L} = \frac{2ZRT\mu\rho v}{Mk} + \frac{2ZRT\beta(\rho v)^2}{M} \quad (3-13)$$

$\frac{p_1^2 - p_2^2}{L}$ is the sum of the viscous loss, $\frac{2ZRT\mu\rho v}{Mk}$, and the inertial loss, $\frac{2ZRT\beta(\rho v)^2}{M}$, where β is the inertial flow coefficient. When the inertial flow term is small, Forchheimer's equation reduces to Darcy's law.

In fracture conductivity experiment, nitrogen flow rate q (liter/min) is measured. As:

$$v = \frac{q}{w \times h} \quad (3-14)$$

Rearrange the equation:

$$\frac{(p_1^2 - p_2^2)Mh}{2ZRTL\mu\rho q} = \frac{1}{k_f w} + \frac{\beta\rho q}{w^2 \mu h} \quad (3-15)$$

This equation can be plotted as a straight line in Forchheimer's graph, $\frac{(p_1^2 - p_2^2)Mh}{2ZRTL\mu\rho q}$ vs. $\frac{\rho q}{\mu h}$. The y intercept is the inverse of the fracture conductivity and the slope is proportional to the inertial flow coefficient. The variables M , T , p_1 , and p_2 are measured in the laboratory. Permeability and inertial flow coefficient are determined simultaneous by drawing the best fit straight line through the data.

The fracture conductivity measurement procedure is:

1. Assemble acid etched core samples into the conductivity cell. Be sure that the fracture interface is lined up with the inlet and outlet flow insert ports. Use a syringe to inject silicone gel into the gaps between the cores and the conductivity cell body. Install the side pistons. The core samples and side pistons fit the conductivity cell tightly. A hydraulic jack is used to push them in position.

2. Put the conductivity cell assembly in the support rack. Adjust the bolts to fit in the load frame. Connect nitrogen hoses from the nitrogen regulator, flow meter to the conductivity cell. Connect the pressure transmitters to the conductivity cell with 1/8 inch propylene tubes and compression fittings.

3. Use a horizontal level meter to make sure that load frame upper plate, the conductivity cell and load frame lower ram are all in horizontal level. Also make sure the nuts on load frame are in tight contact with the upper plate.

4. Run the LabVIEW program “Conductivity pressures.vi” to record the conductivity cell middle point pressure and the pressure drop along the fracture.

5. Activate the AP-1000 hydraulic oil pump by opening the air supply valve. Operate the air pressure regulator and the hydraulic oil pressure regulator to pump hydraulic oil to the load frame. The load frame bottom ram will rise. Watch the gauge pressure increase to a desired fracture closure pressure and let it stabilize for 50 minutes. 95 psi of hydraulic oil gauge pressure is a good start point. It imposes about 1000 psi closure stress on the cores’ fracture face.

Note: The core samples’ fracture face is about 11.9 inch². The ram area of the load frame is 125 inch².

6. Open the nitrogen regulator to about 80 psi outlet pressure. Adjust the nitrogen flow rates in sequence of a set of numbers: 5 L/min, 10 L/min, 15 L/min and 20 L/min. Use the back pressure regulator in the conductivity cell effluent line to adjust the pressure in the middle of the conductivity cell. A 50 psi reading is desired. Let the nitrogen flowrate and the pressures stabilize. Record the stable nitrogen flowrate, the conductivity cell pressure, and the nitrogen pressure drop along the fracture.

7. Adjust the nitrogen mass flow controller to change the nitrogen flowrate. Use the back pressure regulator to keep the conductivity cell pressure the same as the previous one. Record the flowrate, the conductivity cell pressure and the pressure drop along the fracture again. Repeat this step to get the pressure data for four different nitrogen flow rates.

8. Increase the load frame overburden to do fracture conductivity measurement at higher fracture closure stresses. Let the load frame stabilize for about 50 minutes and then repeat steps 6 and 7. The load frame hydraulic oil gauge pressures are increased in sequence: 195 psi, 295 psi, 395 psi, 495 psi, and 595 psi.

9. When all the measurements are done, close the nitrogen regulator and relieve the conductivity cell pressure. Operate the load frame valve to unload the overburden on the conductivity cell. Lower the bottom ram of the load frame and take out the conductivity cell assembly. Disassemble it and get the cores out.

10. Process the data in MS Excel spreadsheet to get the fracture permeability data under each closure stress.

CHAPTER IV

PRELIMINARY EXPERIMENTS AND RESULTS

Several preliminary acid frac conductivity experiments were done with different acid types and limestone cores. The laboratory apparatus is tested to its operating range. The setup is adjusted to better fit its working conditions. The experimental results are recorded and analyzed.

4.1 Experimental parameters

The acid is heated up to 200°F before entering the conductivity cell. The cores are Indiana Limestone and are initially saturated with water. The acid flowrate is about 1 L/minute and the back pressure is set at 1000 psi. The initial fracture gap is set at 0.12 inch.

Four different acid fluids are used:

A: 15% HCl + Corrosion inhibitor

B: 15% HCl + Gelling agent + Methanol + Corrosion inhibitor

C: 15% HCl + Gelling agent + Iron stabilizer + Corrosion inhibitor

D: 28% HCl + Diesel + Emulsifier + Corrosion inhibitor

Fluid A, B and C are mixed with the same concentration of hydrochloric acid. A is straight acid with corrosion inhibitor. It is for lab uses only and is not a formula for oilfield applications. B is a viscosified fluid with methanol. C is a viscosified fluid with a stabilizer. Both B and C are viscous gel, which have effective fluid loss control in carbonate formations. D is mixed from high concentration hydrochloric acid (28%) with diesel and emulsifier. The mixture is a homogeneous emulsion fluid, which also has good fluid loss control. However after pumping through the core fracture, the heated fluid D tends to separate its water phase from the diesel phase. It does not seem to be a homogeneous mixture in the spent acid tank.

For each fluid, we used three different acid contact times with limestone cores: 15 minutes, 30 minutes and 60 minutes. The fluid temperature, fracture width and flux rate are kept constant for all the experiments.

4.2 Preliminary experiments results

The experiments results are shown in the following pictures, spreadsheet, and charts. As shown in the following figures 4.1 through 4.9 in section 4.2.1, the effect of acid contact time is obvious. The longer the acid reaction time, the more rock are dissolved. The carbonate is dissolved heterogeneously at the core surfaces. The wormholes distribute randomly in the cores. We found that different types of acid fluids create different surface etching result:

- Straight acid reacts fast and remove the most carbonate from the cores;
- Emulsified acid reacts the slowest;
- Gelled acids create the most wormholes in the cores.

As we used fluids with different viscosities, we found that the viscosity affects acid transport and heat transfer.

4.2.1 Acid etched core samples pictures



Figure 4.1 Acid A with limestone at 15 min contact time

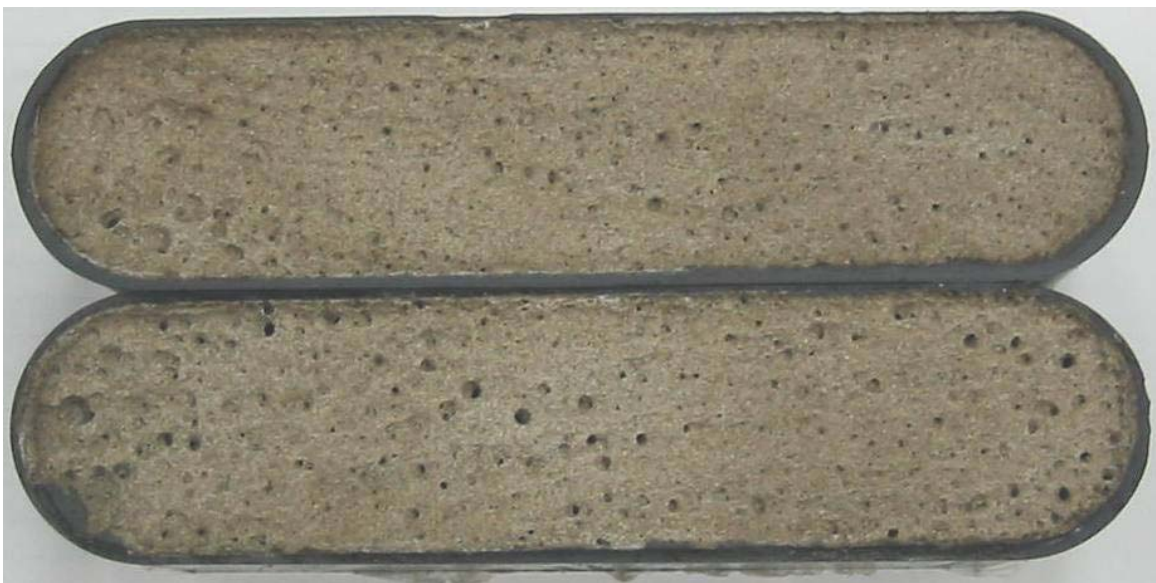


Figure 4.2 Acid A with limestone at 30 min contact time



Figure 4.3 Acid B with limestone at 15 min contact time



Figure 4.4 Acid B with limestone at 30 min contact time



Figure 4.5 Acid B with limestone at 60 min contact time



Figure 4.6 Acid C with limestone at 15 min contact time



Figure 4.7 Acid C with limestone at 30 min contact time



Figure 4.8 Acid C with limestone at 60 min contact time

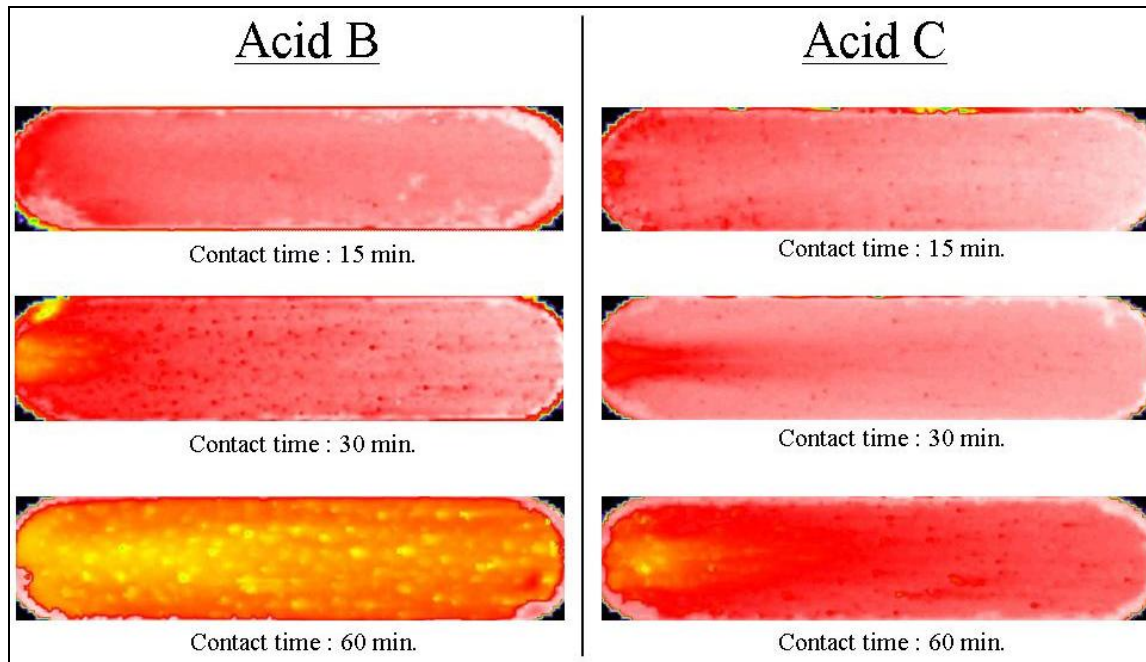


Figure 4.9 Core surface profile comparison

4.2.2 Acid fracture conductivity measurement results

The fracture conductivity data of the acid etched cores are plotted in Figures 4.10, 4.11 and 4.12. The fracture conductivity data do not show any trend corresponding to the acid frac experimental parameters as we expected. It does not repeat the experimental results in Nierode and Kruk's⁷ study. The basic conclusions are:

- Fracture conductivity decreases significant as closure stress increases.
- More uneven core surface should have higher residual fracture conductivity. However, it is not obvious in the charts.
- The conductivity results fluctuate in a wide range.
- Proper experiment operation is critical for valid results.

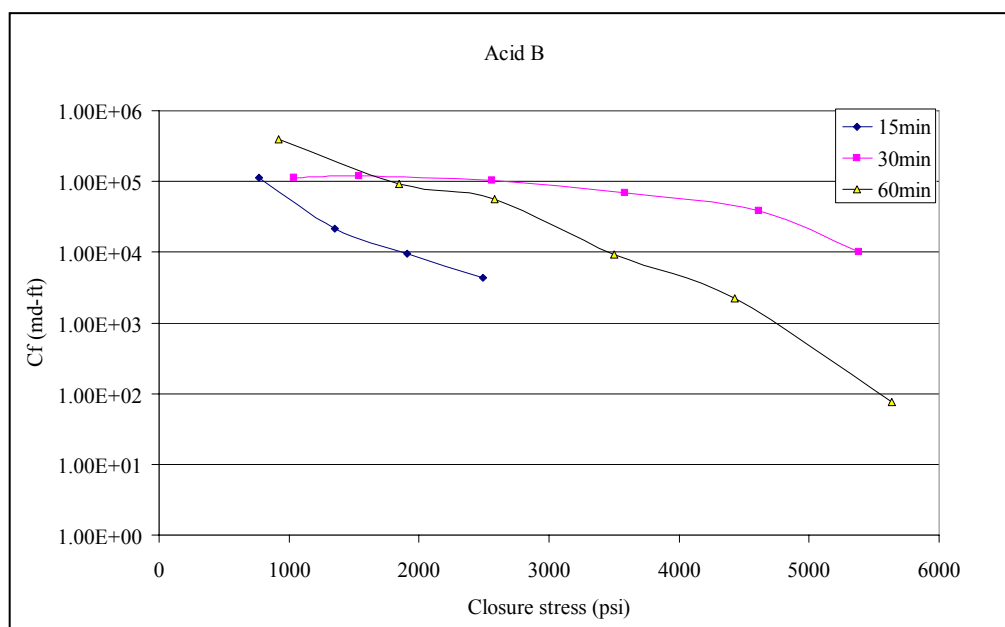


Figure 4.10 Acid B - fracture conductivity chart

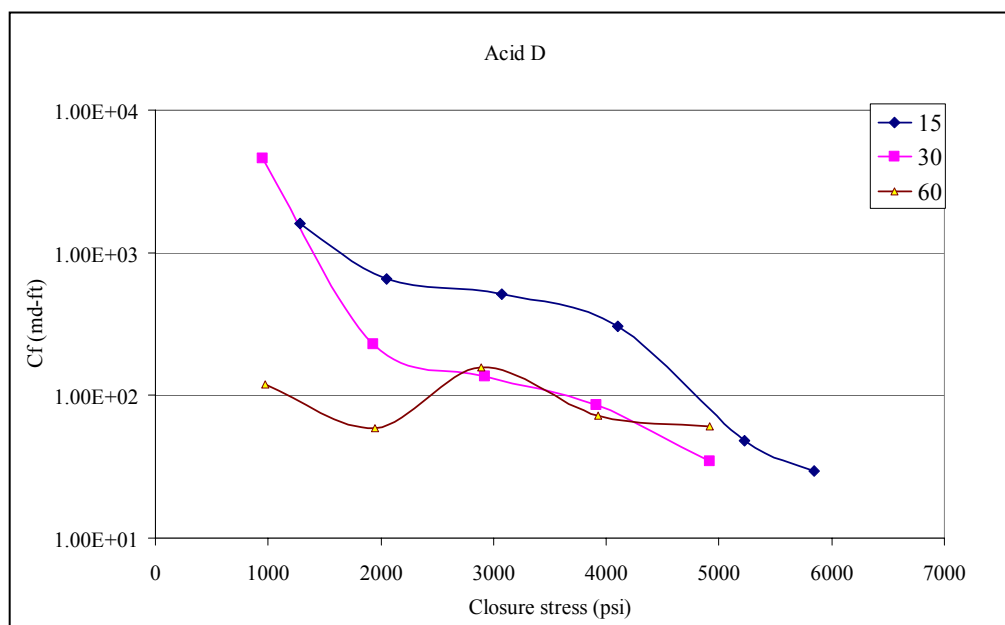


Figure 4.11 Acid D - fracture conductivity chart

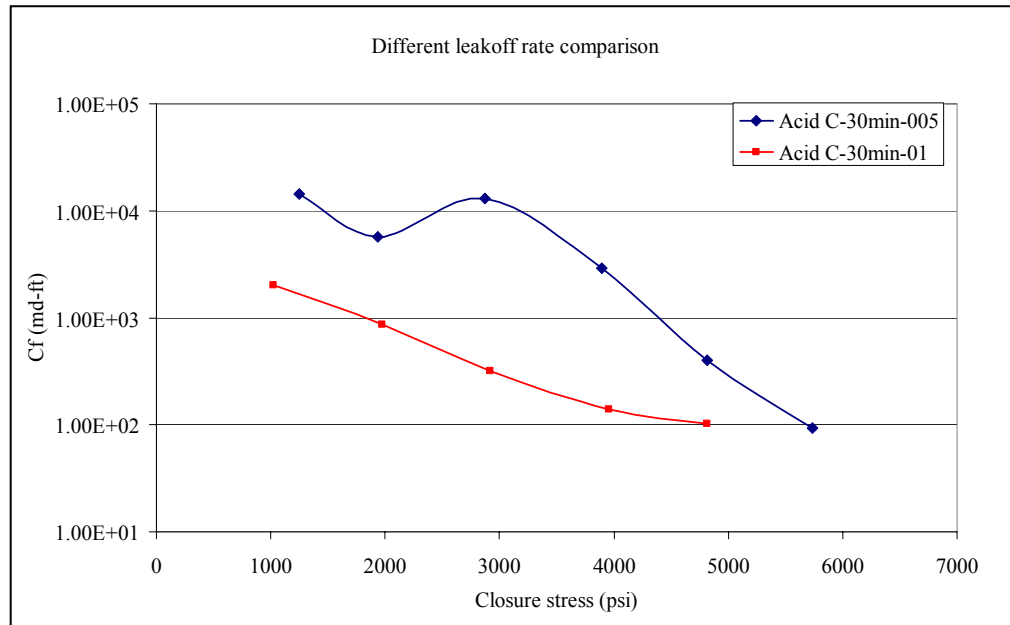


Figure 4.12 Acid C - fracture conductivity chart

4.3 Discussion

1. Some acid treatment fluids require special procedure or shear force to make it work properly. The detail must be observed. The shear force can be achieved with the Maxi-Stirrer.

2. In acid etching experiments, the acid fluid pressure inside the conductivity cell is critical. It has to be high enough to keep carbon dioxide generated from the chemical reactions dissolved in the liquid. Otherwise, the CO₂ bubble will act as a diverter and force acid into core samples pores and cause excessive wormhole, as Figure 4.13: the cell pressure was only 75 psi. 1000 psi pressure is ideal.



Figure 4.13 Acid etched core sample (75 psi back pressure and 23 minutes contact time)

3. The measurement of fracture conductivity requires blocking all possible nitrogen flow bypasses. Our tests show that flow may occur in the gap between the cores and the cell body. It caused our failure in the first two measurements. Silicone gel can help to avoid the problem.

4. The effect of leakoff velocity should be studied. Leakoff affect acid fracture width and length, wormhole size and density. It is one of the most important variables for a successful acid fracture treatment.

CHAPTER V

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The objectives are to develop a new approach to acid fracture modeling and to conduct new laboratory acid fracture conductivity experiments to augment the limited data now available.

The experiments are designed to reflect the acid fracture field conditions. Each experimental variable is studied carefully to make it realistic. The project team did extensive research on acid frac hardware. The selected equipments and instruments meet the researchers' requirements and enable the experiments success.

Some preliminary experiments were carried out with Indiana limestone. Four types of acid fluids are used. The acid mixtures have different viscosity, concentration and fluid loss control additives. Three different acid etching treatment times are experimented, at the same back pressure, temperature, and acid flux rate.

The acid etched core samples were scanned with profilometer. The rough rock surface is digitalized at points every 0.05inch x 0.05inch. The surface unevenness is analyzed with statistics method. The acid fracture conductivity is measured with nitrogen flow.

From the limited acid frac experimental results, some conclusion can be drawn:

1. Treatment fluid effect: Acids of different properties, such as viscosity, concentration, act different with carbonate rocks. The effect has to be further studied. More extensive experiments are required to evaluate the fluids.
2. Acid contact time effect: acid etching results of different acid contact time differ significantly. For all acid types, the longer acid contact time, the more carbonate rock dissolved: bigger fracture width and more wormholes.

3. Wormhole control: leakoff velocity is controlled by back pressure regulator. The pressure drop across the core samples dominates the leakoff flow rate. In the preliminary experiments, leakoff velocity is in the range of 0.003~0.01 ft/min.

5.2 Recommendation for future acid fracture research work

There are still a lot to be improved in acid fracture conductivity experiments. In acid etching treatments, different fracture widths and different acid reaction temperatures are to be tested. In acid fracture conductivity measurement, the accuracy of the results is to be evaluated. It is recommended that running some measurement with water in the conventional way. Compare the results of both methods and find out the cause of the difference, if there is any. Also, it is useful to measure the fluid viscosity for each type of acid mixture, as the acid viscosity may affect the acid etching result.

NOMENCLATURE

A	=	cross-sectional area (cm ²)
N_{Re}	=	Reynold's number
D	=	Diameter
v	=	Fluid velocity (ft/min)
ρ	=	Density (lbm/ft ³)
μ	=	Fluid viscosity (cp)
w_f	=	Fracture width (ft)
h	=	Fracture height (ft)
N_{Pe}	=	Peclet number
D_{eff}	=	Diffusivity coefficient
x_f	=	Fracture length (ft)
q	=	Fluid flow rate (Liter/min)
p_1	=	Upstream pressure (psi)
p_2	=	Downstream pressure (psi)
L	=	Distance between upstream and downstream ports (ft)
Z	=	Nitrogen ... factor
R	=	constant parameter???
T	=	Nitrogen temperature (deg. F)
M	=	Molecular mass (g/mole)

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APPENDIX A

1. Acid etching experiment data sheet sample

0	Run LabVIEW	Rock type: <u>Limestone</u>	Experiment date: <u>2005-11-5,</u>	<u>Afternoon</u>
		Acid type: <u>SLB SXE28%</u>		
1	Rock thickness (inch) and weight (g)			Hardness <u> </u>
	Rock # <u>S8A</u>	Weight (before) <u>127.28</u>	Thickness <u>2.897</u>	
	Rock # <u>S8B</u>	Weight (before) <u>127.4</u>	Thickness <u>2.925</u>	
2	Fracture width setting (inch)	3 Piston position (inch)	- east <u>2.972</u>	
	<u>0.12</u>		- west <u>2.98</u>	
4	Flow meter (Liter)			
	Accumulation reading before pumping <u>---</u>			
5	Acid tank level (Liter)	6 Water tank level (Liter)		
	start <u>82</u>	start <u>90</u>		
7	Temperature (deg. F) <u>200</u>	(fluid inside tube)		
8	Time (min:sec)	There was problem with leakoff back pressure regulator. Changed it before acid.		
	Water start time <u>--</u>			
	Acid start time <u>0:00:00</u>	Total water time <u> </u>		
	Flush start time <u>1:00:00</u>	Total acid time <u>1:00:00</u>		
	End pumping time <u>1:20:00</u>			
9	Stable pressure reading (psi)			
	during water pumping <u>1000</u>	Leakoff volume: <u>1352</u> ml		
	during acid pumping <u>1000</u>	Leakoff flux: <u>0.00500</u> ft/min		
	during water flush <u>1000</u>	Calculated based on leakoff volume		
<u>After Acid Injection</u>				
1	Piston position (inch)	2 Rock weight (g)		Hardness <u> </u>
	- east <u>2.972</u>	Rock # <u>S8A</u>	after <u> </u>	
	- west <u>2.98</u>	Rock # <u>S8B</u>	after <u> </u>	
3	Flow meter reading (start acid) <u>1437.3</u>			
	Accumulation reading at acid end <u>1527.6</u>			
	Total acid pumped (Liter) <u>45.15</u>	(fault number, flow meter needs calibration.)		
4	Acid tank level (Liter)	5 Water tank level (Liter)		
	end <u>20</u>	end <u>62</u>		
	pumped volume <u>62</u>	pumped volume <u>-</u>		

2. Fracture conductivity experiment data sheet sample

Experiment Date: 2005.11.21		Rock Number		
Pressure setting:		Rock # SLB9A		
Inflow pressure on tank	80 psi	Rock # SLB9B		
Backpressure	psi			
Overburden Pressure (psi)	Flowrate (LPM)	Fracture Pressure Drop (psi)	Absolute Pressure (psi)	Temp. (F)
		45 min	45 min	
627	20	0.75	50	
	30	1.57	50	
	40	2.7	50	
	50	3.95	50	
1253	20	3.25	50	
	30	6.64	50	
	40	11.18	50	
	50	15.95	50	
2005	25.5	8.51	50.1	
	30	11.3	50	
	35	14.26	50	
	40	17.32	50	
3007	20.4	8.13	50	
	30	14.52	50	
	40	23.5	50	
	43.9	26.8	49.9	
4010	20.3	15.42	50.1	
	25	19.82	50	
	30	28.2	49.9	
	40	44.9	50	
5112	9	20.9	59.9	
	10.4	26.6	60	
	12	31.2	60.3	
	13.9	38.3	60.1	
5664	7.6	25.5	60	
	9.1	32.5	60.1	
	10.9	41.3	60.2	
	12.8	51.3	60.2	
	13.3	54.1	60	

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